PRECIPITATION PROCESS FOR SOLID SUGAR AND OIL PRODUCTS

Inventor: Lee; John H.; 1441 N. Lucy Montgomery Way Street, Olathe, KS 66061 (US)

Assignee: Rigel Technology Corporation; 11800 W. 63 Street, Shawnee, KS 66203 (US)

Reference Cited:

U.S. Patent Documents

3961081	Jun. 1, 1976	McKenzie	426/658
4138505	Feb. 6, 1979	Hart et al.	426/98
4216234	Aug. 5, 1980	Rawlings et al.	426/98
4217370	Aug. 12, 1980	Rawlings et al.	426/98
4808429	Feb. 28, 1989	Freeman	426/647
5143737	Sept. 1, 1992	Richardson	426/2
5456921	Oct. 10, 1995	Mateescu et al.	426/465
5514388	May 7, 1996	Rohwer	426/231
5874102	Feb. 23, 1999	LaJoie et al.	424/438
5928687	Jul. 27, 1999	Meade et al.	426/2
6153236	Nov. 28, 2000	Wu et al.	426/98
6168803	Jan. 2, 2001	Harris et al.	424/442
6229031	May 8, 2001	Strohmaier et al.	554/156
6368657	Apr. 9, 2002	Lee	426/658
6419957	Jul. 16, 2002	Lanaerts et al.	424/488
6436461	Aug. 20, 2002	Bouwmeesters et al.	426/575
6440478	Aug. 27, 2003	Benton et al.	426/471
6576667	June 10, 2003	Strohmaier et al.	514/560

PRECIPITATION PROCESS FOR SOLID SUGAR AND OIL PRODUCTS

CROSS-REFERENCE TO RELATED APPLICATION: This application is a continuation-in-part application of U.S. application Ser. No. 09/920,125, filed on August 1, 2001, now U.S. Patent No. 6,368,657.

BACKGROUND OF THE INVENTION

Molasses is a liquid sugar by-product, which comes from the processes of cane or beet sugar, corn ethanol, soybean protein and other products. Molasses contains sugar, ash, protein and others. The molasses sugar products are often concentrated into a viscous and sticky liquid at solids such as 50-80 % by a vacuum heat process. It is very difficult to convert the liquid molasses products into solid products in particle form without the effects by heat and moisture. Molasses becomes much more soft by heat. Also molasses dissolves into water. For feed applications, molasses is often used for liquid feeds, animal blocks and as a general feed ingredient. For animal feed block supplement, the block product is a large block piece, which is described by U.S. Patent Nos. 6,440,478, 6,168,803, 5,755,178, 5,622,739 and 3,961,081. Lactose whey is a liquid sugar by-product, which comes from cheese, whey protein or pure lactose processing. Milk contains lactose, casein and whey proteins. After cheese processing, the mixture of whey proteins and lactose is processed by an ultra filtration to separate into whey protein fraction and lactose fraction. Pure lactose product is produced from the lactose fraction. Lactose whey has similar characteristics as molasses. The concentrated lactose whey is a viscous and sticky liquid. Lactose whey has a light color. Molasses has a dark color. U.S. Patent 6,368,657 discloses a precipitation process to convert liquid molasses or lactose whey with liquid animal blood into solid products in particle form without the effects of heat and moisture. But the product has dark color. Also there is no fat in the solid products. This invention has further developed more practical products with better functions. Besides the solid sugar-protein products, new solid sugar-protein-fat and sugar-fat products have been developed. Fat increases the energy value for feed products. The new solid sugar-protein-fat products improve the fat and molasses properties, which are not effected by heat and moisture. Fat is a liquid oil form under heat. The process in this invention also converts the liquid oil into solid fat. The products are improved with lighter color and mild smell by peroxide treatment.

Vegetable soap stock such as soy, corn or canola oil soap stock is the vegetable oil by-product during vegetable oil process. Hydrolyzed vegetable or animal oil is obtained after hydrolyzed processes such as hydrolyzed fish or tallow oil. It has been found by this present invention that vegetable or animal soap stock or hydrolyzed oil is used as an oil ingredient into the liquid sugar and protein to be processed to form solid sugar-protein-fat precipitation products without oily and sticky physical properties. The new solid precipitation products can withstand the heat and moisture without changing product properties, which are totally different from normal molasses or oil products. The products can be either wet or dry solid form. Also vegetable soap stock such as soy, corn or canola oil soap stock can be converted into new solid precipitation products with such as agar, cellulose, milk, casein, whey, plasma, egg or soy protein. When a blender such as homogenizer or high speed agitation is used, the wet soft solid protein-fat product is easily broken into very fine pieces and the liquid with the fine pieces is dried by a spray dryer into powder form.

Over the years, various attempts have been made to make solid molasses or fat products. A number of patents have been issued to make the products by some special treatments. U.S. Patent No. 6,576,667 discloses a process method to manufacturing fatty acid calcium salts in solid form from high glyceride content oils. U.S. Patent No. 6,440,478 discloses a process to produce an improved feed molasses block in which a fluid mixture comprising molasses, additives and other nutrients into a dehydration vessel operating under a partial vacuum. Once an appropriate amount of water has been removed, the dehydrated fluid mixture is formed into block shape and cooled to form feed solid blocks. U.S. Patent No. 6,436,461 discloses a process to prepare the gel beads containing acid polysaccharide (carbohydrate) and voltaile liquid ingredient by forming of discrete droplets and converting the droplets into water-insoluble gel beads by introducing the droplets in a solution with multivalent cations. The ingredient consists flavors, fragrances, vitamins or coloring materials. U.S. Patent No. 6,229,031 discloses a process method for the preparation of fatty acid calcium salt, which can be used as rumen bypass feed supplements. Fatty acid, calcium oxide and water is heated to a temperature at which the fatty acid glycerides saponify to form fatty acid calcium salt. U.S. Patent No. 6,153,236 discloses a process for low melt encapsulation with high laurate canola oil. U.S. Patent No. 5,928,687 discloses a rumen by-pass feed supplement in the form of a matrix containing biologically active material, protective substance, and surface coating impervious to attack in the rumen. U.S. Patent No. 5,874,102

discloses a process to prepare a dietary fatty acid salt product in granulated form, which can function as a rumen bypass animal feed supplement. U.S. Patent No. 5,514,388 discloses a process to treat protein with a base to increase the pH to such as 12 at first. Then the fat is added into the alkali protein. The protein firm gel product is formed when the pH is lowed to 3 to 5. U.S. Patent No. 5,143,737 discloses a process method to produce unsaturated milk fat or meat fat to be encapsulated with an acid sensitive nontoxic crosslinkable material. U.S. Patent 4,808,429 discloses a process to encapsulate animal blood and fat by heating to a temperature in the range 40-45 degree C and homogenizing into a dispersion or emulsion. Then the mixture was allowed to cool for forming a gel product and then dried. U.S. Patent No. 4,217,370 discloses a process to treat protein with a base to increase the pH such as 11 at first. Then the fat is added into the alkali protein. The protein and fat are mixed together. The protein-fat gel product is formed when the pH is lowed to the isoelectric point. U.S. Patent 4,216,234 discloses a process to render albumin containing medium with strongly alkaline (e.g. pH 11) prior to the formation of the fat dispersion or emulsion. The fat is encapsulated to form a gel-form product. U.S. Patent 4,138,505 discloses a process to heat animal blood to a temperature in the range of from about 20 to about 60 degree C, to adjust the pH of the heated aqueous medium to a strongly alkaline such as pH 12 to form a blood solid gel, and thereafter recovering the fat-protein gel. U.S. Patent No. 3,961,081 discloses a method to prepare a solid molasses feed block for animals. The block embodies molasses solids as a primary structural and nutritional constituent, which provides the ingredients and advantages of a liquid feed supplement in solid form.

SUMMARY OF THE INVENTION

The present invention provides the precipitation process to form solid sugar-protein and sugar-protein-fat precipitation products.

For preparing solid sugar-protein or sugar-protein-fat precipitation products, liquid molasses or lactose whey is mixed with liquid animal blood, which is animal whole blood or red blood cells such as from pork, beef and poultry. If an oil ingredient such as soy or corn soap stock is added into the liquid sugar by-product and protein ingredients, the new solid product is processed to have a fat content such as 30 % without oily physical characteristics. A normal product with the

the soap stock at a fat content such as 30 % is very oily. Some chemical such as calcium or manganese in oxide or hydroxide form is added for combining more fatty acid oil to form the soap under heat treatment into the solid product. The value of pH is checked and adjusted. Then a direct or non-direct heat source is applied to raise the temperature to above 60 degree C to convert liquid molasses or lactose whey and liquid animal blood into the precipitation solid product by the protein coagulation. If peroxide is added into the wet product, the color is changed to a lighter color such as brown or yellowish compared with the natural black-reddish color of dried animal blood meal without peroxide treatment. The color change depends on the added level of the peroxide. The free water in the wet solid product is removed by a press, screen or centrifuge to reduce the drying cost if there is free water. Then the wet solid product is dried by a dryer. The solid precipitation product has no sticky physical characteristics. Normal wet molasses is always a liquid form under heat. The molasses at very low moisture level can form solid product after cooling down, which becomes soft under heat and dissolves into water. This process changes the molasses properties. There are no similar effects by heat and moisture to the new solid precipitation product, which is totally different from normal molasses or oil products. Lactose whey is used similar to molasses. Besides dry solid product with longer shelf time, the wet solid product may be used in some applications. Peroxide treatment improves the product with light color and mild smell, which is from hydrogen peroxide, calcium peroxide or sodium peroxide. The treatment increases the coagulation and improves energy consumption in the heat process, waste water situation, and micro counts.

Vegetable soap stock such as soy, corn or canola oil soap stock, which is the vegetable oil by-product produced during vegetable oil process, or hydrolyzed oil is easily mixed into liquid sugar by-product and liquid animal blood and heated to form the new solid sugar-protein-fat precipitation product by the protein coagulation. The new solid precipitation product has no oily and sticky physical characteristics. Also new precipitation soap stock products are formed with some proteins or carbohydrates after coagulation, which have no oily physical characteristics. Besides as a general nutritional ingredient, the protein-fat or sugar-protein-fat precipitation products can be used as by-pass ingredients for dairy animals to produce more milk. The reason may be the hydrophilic groups of vegetable or animal soap stock or hydrolyzed oil bind to wet protein or/and carbohydrate ingredients and then the hydrophobic groups of the soap stock or hydrolyzed oil are encapsulated during the protein or carbohydrate coagulation process to form

the precipitation product without oily physical characteristics. Some protein and carbohydrate ingredients have the coagulation function under temperature or pH change (to higher or lower) treatment such as milk, casein, whey, soy, plasma, gluten, egg, and rice protein, agar, molasses, starch, and cellulose besides animal whole blood and red blood cells.

Fat provides energy for animals. Molasses and other sugars provide sweet taste. So new solid sweet protein, sweet fat (energy) or sweet protein and fat (energy) products can be made by this invention. For sweet fat product, liquid sugar such as molasses is mixed with animal or vegetable oil at high free and saturated fatty acids or saturated fatty acids such as more than 30 % and heated the mixture into a uniform liquid at low moisture level (less than 15 %), then the mixture is sprayed and cooled or cooled and ground into solid particles. Most animal or vegetable oil has low saturated fatty acids and does not work. Vacuum process may be used to remove moisture quickly. Saturated fat has high melting point. Molasses coagulates into solids during cooling. The new solid sweet fat product should have good potential in different feed applications. Some cross-linking agents such as calcium and formaldehyde help the encapsulation.

For organic chemicals such as some medicines or flavors, the hydrophobic groups of the organic medicine or flavor bind with the hydrophobic groups of an organic solvent by hydrophobic bonds after the organic solvent is mixed with the organic medicine or flavor. The organic solvent needs to have both hydrophobic and hydrophilic groups. The hydrophilic groups of the organic solvent bind to protein or carbohydrate ingredient and then the hydrophobic groups of the organic solvent and the organic medicine or flavor are encapsulated by the protein or carbohydrate coagulation process with temperature or pH change. Hydrolyzed vegetable or animal oil or soap stock can be used as one solvent with both hydrophobic and hydrophilic groups. The hydrolyzed oil products such as from soybean, sunflower seed, corn, coconut, safflower seed, cotton seed, palm, and canola oil ingredients fit the encapsulation process in this invention. There are other solvents with both hydrophobic and hydrophilic groups. If the organic medicine or flavor itself has both hydrophobic and hydrophilic groups, the organic solvent is not necessary to be needed for encapsulating the organic medicine or flavor. The encapsulated organic medicine or flavor has the function to be released slowly in human or animal bodies. The medicine release from the biodegradable protein or carbohydrate polymer is dependent on the polymer erosion to affect the delivery system in the bodies. The medicine is released slowly. This encapsulation process in this

invention is more practical for large-scale production than the cross-linked carbohydrate amylose or cellulose product with even toxic cross-linking chemicals to form a matrix for the slow release of medicine agents, which are described in such as US Patents 6,419,957, 6,284,273, 5,989,589, 5,807,575, and 5,456,921. The present precipitation process is a more economic and practical process, which may be used to make the encapsulation process easily commercialized.

DETAIL DESCRIPTION OF THE PREFERRED EMBODIMENTS

The following examples set forth preferred methods in accordance with the invention. It is to be understood, however, that these examples are provided by way of illustration and nothing therein should be taken as a limitation upon the overall scope of the invention.

EXAMPLE 1

Liquid beet molasses (50 grams at 80 % solids) was mixed with liquid pork red blood cells (92 grams at 30 % solids). Then mixture was mixed and heated to 85 °C for 1 minutes. The wet precipitation product mixture was dried in an oven at 105 °C for 4 hours. The solid product was easily broken into small particles. The analytical data were follows: protein (39.8 %), moisture (7.8 %), and ash (8.3 %). The product had no sticky physical characteristics.

EXAMPLE 2

Liquid cane molasses (16 grams at 79 % solids) was mixed with soy hydrolyzed oil (17 grams processed from soy soap stock) and heated to 55 °C and then mixed with calcium oxide (0.5 grams) and liquid pork red blood cells (68 grams at 30 % solids and 30 °C). Then mixture was mixed and heated to 75 °C for 0.5 minutes. Then 2 ml of 30 % hydrogen peroxide was added into the wet precipitation product and mixed. The wet product was dried in an oven at 105 °C for 5 hours. The solid product was easily broken into small particles. The analytical data were follows: fat (30.1 %), protein (41.6 %), moisture (6.9 %), and ash (8.8 %). The product had no oily and sticky physical characteristics.

EXAMPLE 3

Liquid lactose whey (30 grams at 45 % solids) was mixed with and liquid pork whole blood (42

grams at 20 % solids). Then mixture was mixed and heated to 85 °C for 0.5 minutes. Then 1.5 ml of 30 % hydrogen peroxide was added into the wet precipitation product and mixed. The wet product was dried in an oven at 105 °C for 5 hours. The analytical data were follows: protein (36.7 %), moisture (5.2 %), and ash (8.6 %). The product had no stick physical characteristics.

EXAMPLE 4

Liquid cane molasses (29 grams at 79 % solids) was mixed with soy soap stock (45 grams at 50 % solid) and heated to 50 °C and then mixed liquid pork red blood cells (105 grams at 30 % solids and 30 °C). Then mixture was mixed and heated to 80 °C for 1 minutes. Then 3 ml of 30 % hydrogen peroxide was added into the wet precipitation product and mixed. The wet product was dried in an oven at 105 °C for 5 hours. The solid product was easily broken into small particles. The analytical data were follows: protein (46.6 %), fat (32.3 %), moisture (7.7 %), and ash (6.8 %). The pepsin digestibility for protein was 95.6 %. The product had no oily and stick physical characteristics. The product had the by-pass protein (96.3 %) and by-pass fat (38.5 %) by the incubation in rumen fluid for 24 hours and the by-pass protein (89.2 %) and by-pass fat (38.4 %) by the incubation in rumen fluid for 48 hours.

EXAMPLE 5

Flavor in hydrolyzed vegetable oil (30 grams) was mixed with egg white (85 grams) and liquid milk (150 grams). The mixture was blended and heated from 25 to 82 °C. The liquid was uniform without oily feeling. The analytical data were follows: solid (22.2 %), protein (5.8 %), and fat (11.0 %).

EXAMPLE 6

Wet agar (164 g) was mixed with soy soap stock (120 g), and Rigel 1010 (0.5 ml). The mixture was dried in an oven at 105 °C for 4 hours. The analytical data were as follows: moisture (6.2 %), fat (33.8 %), and ash (5.1 %). The product had no oily physical characteristics.

EXAMPLE 7

Soy protein concentrate (100 grams at 10 % solid) was mixed with soy soap stock (15 grams at 50 % solid). Then mixture was blended into a uniform liquid. The pH of the mixture was

changed to 3.0 with 10 % hydrochloric acid. The uniform liquid was dried in an oven at 105 °C for 7 hours. The solid product was ground into small particles. The analytical data were follows: fat (36.2 %), protein (35.1 %), moisture (5.8 %), and ash (7.3 %). The product had no oily physical characteristics.

EXAMPLE 8

Liquid beet molasses (60 grams at 80 % solid) was mixed with partially hydrogenated pork fat (48 grams at 99 % crude fat and >80 % saturated fatty acids) and heated to 100 °C for 8 minutes. The uniform liquid mixture was dropped into the pieces at about 0.5 to 1 cm length. After setting and cooling at room temperature for 2 hours, the mixture became the solid product. The solid sugar-fat product was ground into small particles by a coffee grinder. The analytical data were follows: fat (48.2 %), protein (5.2 %), moisture (5.9 %), and ash (4.3 %).

EXAMPLE 9

Soy soap stock (50 grams at 50 % solid) was mixed with liquid pork red blood cells (85 grams at 30 % solids and 30 °C). Then mixture was mixed and heated to 80 °C for 0.5 minutes. The wet product was dried in an oven at 105 °C for 5 hours. The solid product was easily broken into small particles. The analytical data were follows: fat (35.2 %), protein (45.1 %), moisture (5.6 %), and ash (5.7 %). The product had the by-pass protein (96.2 %) and by-pass fat (62.7 %) by the incubation in rumen fluid for 24 hours and the by-pass protein (86.6 %) and by-pass fat (59.1 %) by the incubation in rumen fluid for 48 hours. The product had no oily physical characteristics.